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Pulsed current measurement of the resistive transition and critical current in high T_c superconductors

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A pulsed current technique for measuring the critical transport properties of superconductors as a continuous function of current or temperature is described. Greatly reduced sample heating permits investigation of both the "weak link" and intrinsic properties of granular superconductors. Results obtained for various high T_c oxide superconductors provide an example of the technique's application. Both intergranular and intragranular critical current densities are extracted from the data. Current-voltage measurements using the pulse method are corroborated by traditional dc measurements.

INTRODUCTION

The standard technique for measuring the critical current I_c of a superconductor[1] consists of applying a constant or direct current (dc) until the voltage V which appears across the sample exceeds a given value. The current I at this point is operationally defined as the critical current. The value of I_c determined from such an experiment can depend on the voltage (or electric field or resistance) criterion chosen.[2] This dc method, although widely used, has the following drawbacks: (1) I^2R heating of the sample and contacts (with total resistance R) can give a misleading low value for I_c, (2) no information is obtained on the rest of the superconducting-to-normal transition, only on the onset of resistance, and (3) special sample mounts (heavy wires, etc.) are needed to carry the large direct currents involved in measurements on bulk samples.

These difficulties can be overcome by (1) placing the sample in good thermal contact with a large heat reservoir (such as a block of copper), (2) applying current to the sample in short pulses at low frequency (reducing the I^2 part of I^2R by the pulse duty cycle factor), (3) using very low resistance current contacts (minimizing the R part of I^2R), and (4) using a small sample cross section (so that a small I gives a large current density J, again reducing the I^2 portion of I^2R). The pulsed current not only greatly reduces the power going into the sample, but also gives the sample time to equilibrate with the reservoir during the time between pulses (when no current flows through the sample).

Pulsed current techniques have been used in the past, but for the most part these efforts have used specially designed circuits for producing high-amplitude, multi-step current pulses,[3] or have been limited to single pulses measured from an oscilloscope screen. More recently, Goldschmidt has described a quasi-dc "pulse" technique[4] in which current is switched on for about 0.5 s, and then turned off for about 5 s to allow for heat dissipation. The method described below uses common, readily available electronic equipment to produce and measure short pulses at a high enough frequency to allow continuous measurements either as a function of current or of temperature. A boxcar averager improves the signal-to-noise ratio by averaging over a number of pulses. The technique, briefly described previously,[5,6] has been used to measure the electrical transport properties of a variety of high temperature ceramic superconductors.

I. PULSED CURRENT TECHNIQUE

The pulsed current method is essentially a standard 4-probe dc measurement of resistance, but with current applied in short pulses at a low frequency. The sample voltage V is measured during these short pulses of current, giving a sample resistance $R = V/I$. The heat generated in the sample is reduced, compared to that produced in a dc measurement, by the duty cycle of the applied pulses:

$$\text{duty cycle} = 100\% \times \frac{(\text{pulse width})}{(\text{pulse repetition period})}.$$

This reduction in sample heating is particularly important for very small samples in which the current contacts are in close thermal proximity to the voltage contacts, or for measurements (such as the temperature dependence of I_c) in which the sample is not immersed in a liquid cryogen.

The experimental setup is shown schematically in Fig. 1. Pulses produced by the pulse generator (Tektronix PG505) are amplified by a high-current power supply (Kepco BOP36-5M or Kepco ATE25-40M, depending on the current range). The boxcar averager (Princeton Applied Research, or PAR, 162) has two gated integrators (PAR 166) designated as A and B. The voltage $V_{I\,\mathrm{read}}$ measured by the boxcar averager at input B during the current pulses yields the current $I = V_{I\,\mathrm{read}}/R_{I\,\mathrm{read}}$. The sample voltage is sent to the differential amplifier (PAR 116 preamplifier for a PAR 126 lock-in amplifier) whose single-ended output is connected to the boxcar averager input A. The sample current and voltage signals going to the boxcar are also monitored on an oscilloscope. Typical waveforms for a $YBa_2Cu_3O_x$ sample are shown in Fig. 2. This sample has a transition midpoint of 92 K, and a normal-state resistance at 93 K of 15.8 mΩ.

If the high current power supply is operating in a constant voltage mode rather than a constant current mode

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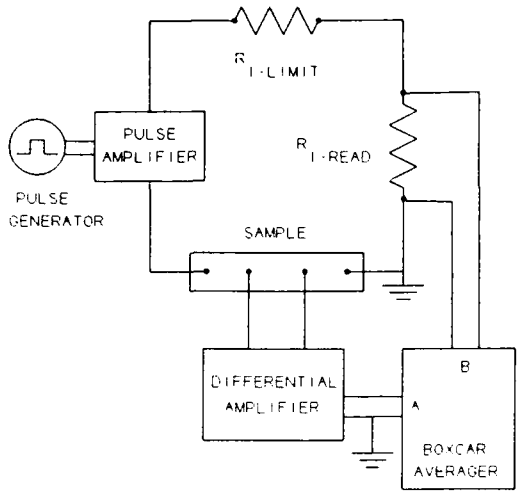

FIG. 1. Schematic of pulsed current circuit.

("constant" during the pulses), then a current-limiting resistor $R_{I\text{-limit}}$ is included in the circuit as shown. The current-read resistor $R_{I\text{-read}}$ is chosen based on the current level used such that the voltage across $R_{I\text{-read}}$ is easily measured. Typical values for this resistor are 1 Ω for $I = 5$ A and 10 mΩ for larger values of I. The value of $R_{I\text{-read}}$ is accurately determined by a dc, 4-probe measurement. The power ratings of $R_{I\text{-limit}}$ and $R_{I\text{-read}}$ must be sufficient to handle the pulsed currents used.

The location of circuit ground has been selected as shown in Fig. 1 both for convenience and to minimize sample voltage noise. Placement of ground between $R_{I\text{-read}}$ and one of the sample's current contacts allows single-ended input of $V_{I\text{-read}}$ to the boxcar averager. Proximity of ground to the sample voltage contacts also helps prevent overloading of the differential amplifier. Note, however, that this choice of ground requires that both the pulse generator and amplifier have a floating output. An isolation transformer between the pulse generator and the power supply could be used to allow one side of the pulse generator output to be grounded. Another alternative is to differentially measure $V_{I\text{-read}}$ before sending the signal to boxcar input B.

It has been found that current pulses with very sharp rise and fall times (< 1 μs) lead to voltage spikes which overshoot and undershoot, respectively, the programmed voltages. These spikes, originating from the pulse generator, quickly relax back in an under-damped oscillation to the expected voltage levels. The spike amplitude increases as the sample goes from the normal to the zero resistance state, which tends to overload the differential amplifier. The effect is merely an artifact of the circuit parameters, having nothing to do with the superconductivity of the sample, and occurs even if the sample is replaced by a short. The deleterious effects of these voltage spikes are minimized as follows. First, the boxcar gate is positioned so that the boxcar "sees" only the interior of the pulses, away from the spikes. Second, the rise and fall times of the pulses are increased to about 10 μs, which greatly reduces the spike amplitude. Finally, the spike amplitude is also greatly reduced by using twisted-pair current leads on the sample probe, including the sample end of the circuit. Standard 50 Ω impedance coaxial cables are used in the room-temperature part of the circuit.

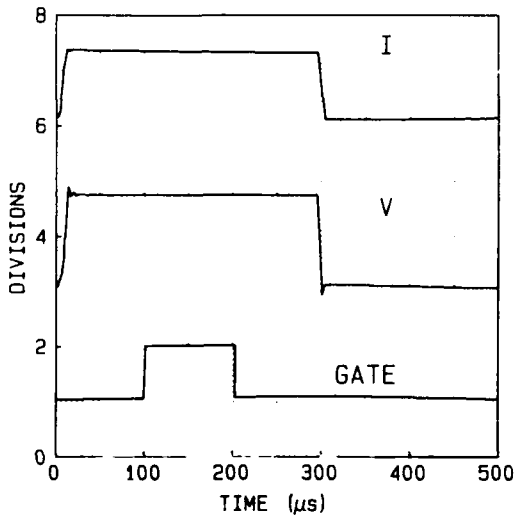

FIG. 2. Digitized photograph of an oscilloscope screen showing current (I, 0.5 A/division), voltage (V, 0.5 mV/division), and gate pulses for a $YBa_2Cu_3O_{7-x}$ sample at a temperature of 84.9 K.

II. APPLICATION TO HIGH T_c SUPERCONDUCTORS

This pulsed current technique has been applied to bulk samples of high T_c superconductors such as $YbBa_2Cu_3O_{7-x}$, $YBa_2Cu_3O_{7-x}$,[7] $Bi_2Sr_2CaCu_2O_x$, and $Bi_2Sr_2Ca_2Cu_3O_x$. The samples were made in the form of flat disks using standard solid state sintering methods, and mechanically cut down to a small cross section.

Two configurations of samples with small cross sections have been used. In the first configuration,[7] four low resistance contact pads (two large current pads and two small voltage pads) are applied to the sample (as described below), which is then glued to an anodized aluminum substrate. Using a $\frac{3}{4}$-in.-diam diamond-coated wheel, a slot is cut down the middle of the disk, cutting completely through the thickness of the sample. The slot is cut almost completely across the face of the disk, leaving only a very small cross section bridge between the two halves of the disk. The slot separates one pair of current and voltage pads from the other pair. The voltage pads are on the bridge end of the sample. This bridge configuration has the advantage of providing large area current pads, giving a very small contact resistance. However, the bridge is very fragile and its cross sectional area is difficult to measure accurately. The accuracy is about $\pm 5\%$ using the traveling stage of a microscope to measure the height and length of the bridge. Another disadvantage is that the length of the bridge (defined by the thickness of the cutting wheel) is only about 1 mm, which means that the voltage measured is smaller than it would be for a longer sample. This effectively decreases the sensitivity obtained at the onset of resistance, thus requiring the use of a higher critical current criterion.

In the second type of sample configuration, the sample

disk is sanded down to a thickness of about 1 mm. Narrow bars about 1 mm^2 in cross section are cut from the disk using the diamond-coated wheel. The cross sectional area is measured with a micrometer to an accuracy of about $\pm$ 1%. Four low resistance contacts are applied along the length of the sample on one of its faces. The sample is then glued to an anodized aluminum substrate. This bar configuration overcomes the disadvantages of the bridge configuration, but at the expense of smaller current contact areas.

Low resistance contacts to the sample are made by applying a thin layer of silver paint (DuPont 4922) in the desired electrode pattern. The painted sample is then placed back in the furnace and heated for approximately two hours at a temperature somewhat below the original sintering temperature. Typical contact processing temperatures are 875 °C for $YBa_2Cu_3O_{7-\delta}$ and 840 °C for $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4+\delta}$. For both materials, the cooldown conditions from high temperature, and any subsequent anneals, are the same as the original processing cycle to insure that the sample characteristics do not change. These conditions include processing the $YBa_2Cu_3O_{7-\delta}$ samples in oxygen and the $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4+\delta}$ samples in air.

Once the low resistance contacts are in place and the sample has been configured as described above, short pieces of solid copper wire are indium soldered to the silver pads. These wires are large enough to carry the maximum current used (keeping in mind the pulse duty cycle) but yet small enough to avoid large heat leaks to the sample. The aluminum substrate, to which the sample is bonded, is attached to the copper sample block of a closed-cycle He-4 refrigerator or of a cryogenic probe using a thin layer of Apiezon-N grease. The temperature of the copper block is measured by a calibrated silicon diode embedded in the block. There is, however, always a small thermal gradient which develops between the sample and sample block during warming or cooling. This temperature difference is monitored continuously by a differential copper-Constantan-copper thermocouple mounted with one junction on the block and one junction on the sample. The sample temperature is then the temperature indicated by the calibrated diode corrected by the temperature gradient measured with the thermocouple. This correction, made in real-time for each data point taken, is usually less than 2 K.

These pulsed current measurements can be performed in two different modes. The first mode is with a constant current pulse amplitude. The sample voltage is measured as the sample slowly warms. This is effectively a measure of sample resistance R versus temperature T, and is used to study the main superconducting transition as well as the development of the resistive "tail" that occurs at high current densities. The results for a $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ sample measured at various current densities in this way are shown in Fig. 3. The pulse width for these measurements was 100 μs and the duty cycle ranged from 0.01 to 0.1%. Another mode of measurement, usually used to study the onset of resistance, is the more familiar I-V measurement, performed at constant temperature, in which the sample voltage is measured as the current pulse amplitude is slowly increased. A series of I-V (actually, current density J vs electric field E) curves at different temperatures is shown in Fig. 4 for the $YBa_2Cu_3O_{7-\delta}$ sample of Fig. 2. The sample cross sectional area is 0.75 mm^2, and the voltage probe separation is 2.6 mm. A pulse width of 300 μs and duty cycle of 0.4% were used for these measurements. Included in Fig. 4 is a pair of curves measured at the same sample block temperature, one using pulsed current and the other using direct current. Essentially the same critical current is obtained from both techniques. As expected, however, the dc value is slightly less than the pulsed value due to increased local heating of the sample. That is, the sample temperature is slightly higher during the dc measurement than during the pulsed measurement, and so the measured critical current is less (see temperature dependence in Fig. 5).

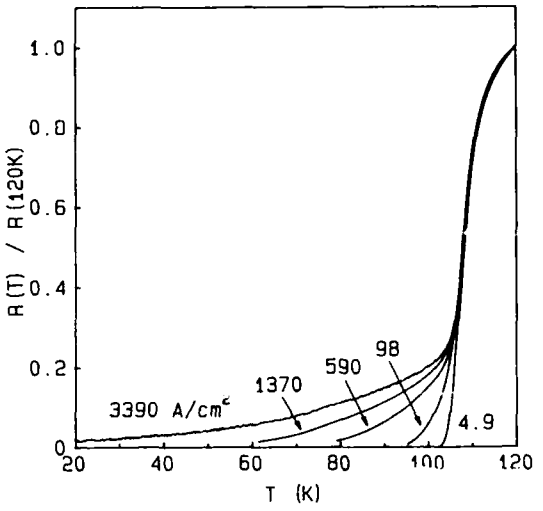

FIG. 3. Normalized resistance of a $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ sample using pulse method at various current densities. Sample resistivity at 120 K is 1.1 mΩ cm.

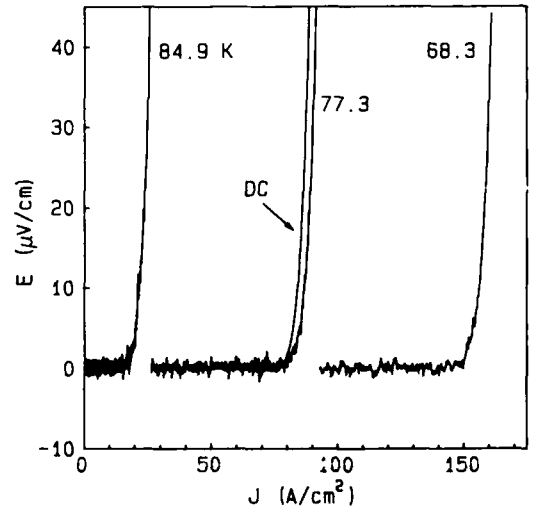

FIG. 4. Current density J vs electric field E at various temperatures for a $YBa_2Cu_3O_{7-\delta}$ sample. The curve marked "dc" was measured with direct current, while the others used pulsed current.

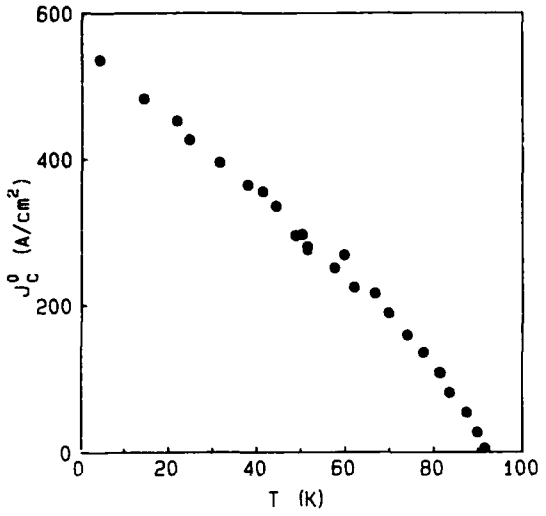

FIG. 5. Temperature dependence of J_c^0 for a $YBa_2Cu_3O_x$ sample measured with the pulse technique.

Two different characteristic critical current densities, corresponding to the two measurement modes described above, can be associated with the granular, bulk form of these high T_c superconducting ceramics.[6,9] The critical current density normally derived from I-V measurements, J_c^0, is characteristic of the weak links which connect the individual, single-crystal grains of superconductor.[10] The temperature dependence of J_c^0, measured with this pulsed current technique, is plotted in Fig. 5 for another $YBa_2Cu_3O_x$ sample. The much higher intrinsic critical current of the grains can also be measured with the same pulse technique. This current density, denoted J_c^{mid}, is obtained by observing the current-induced suppression of the midpoint of the main superconducting transition. J_c^{mid} is plotted in Fig. 6 as a function of the transition midpoint temperature for the sample of Fig. 5. Extrapolation to lower temperatures gives values of intrinsic critical current density which are in very good agreement with results obtained from other techniques, such as magnetic hysteresis experiments.[6]

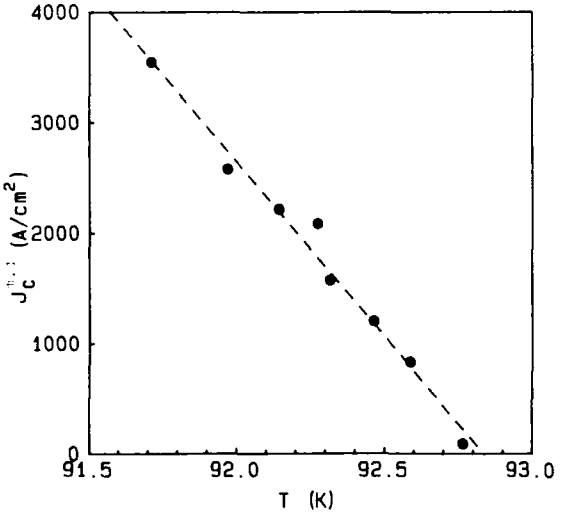

FIG. 6. Temperature dependence of J_c^{mid} near the main superconducting transition for a $YBa_2Cu_3O_x$ sample measured with the pulse technique. An extrapolation of the dashed line (least square fit) gives an intrinsic critical current density at 77.3 K of 4.9×10^4 A/cm^2.

At a high enough pulse duty cycle, or for a resistive enough sample or large enough current-contact resistance, sample heating can be a problem, just as in the dc measurement case. The most direct indication of heating is an increase in the temperature difference ΔT between the sample and the copper block (over and above the usual thermal lag between the two) as measured by the differential thermocouple. Although a constant ΔT is the most desirable, an increase of a few degrees is acceptable as long as it is taken into account in determining the sample temperature. Only a few of the pulsed current measurements made at this laboratory on high T_c superconductors have shown heating to this extent. Another way to gauge the effects of sample heating is to do a measurement at a given current and duty cycle, and to then compare these results to those obtained when the duty cycle is increased (say by doubling the pulse frequency), but with the same current. The measured result should remain the same if sample heating is insignificant. One or both of these tests can be used to select an appropriate pulse duty cycle when setting up the experiment.

A natural extension of this pulse technique is the measurement of the superconducting properties of high T_c thin films. Very large current densities can easily be produced because of the small cross sections of the films. In addition, critical current and resistance measurements as a function of magnetic field should be quite practical for both bulk and film samples.

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